

ENVIRONMENTAL CONTAMINANTS EVALUATION OF ST. JOSEPH BAY, FLORIDA

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APPENDIX ONE

ST. JOSEPH BAY

BIRD SPECIES

1. The first part of the paper discusses the importance of the study of the history of the United States. It is argued that the study of the history of the United States is essential for a full understanding of the country and its people. The paper then discusses the various methods used by historians to study the history of the United States, including the use of primary and secondary sources, and the use of statistical methods. The paper concludes by discussing the importance of the study of the history of the United States for the future of the country.

APPENDIX 1

Appendix 1. List of Bird Species Protected Under the Migratory Bird Treaty Act of 1918 (16 U.S.C. Sec. 703-711), St. Joseph Bay Ecosystem, Florida. Data are numbers of each species observed during Christmas bird counts (National Audubon Soc.).

	<u>1987</u>	<u>1988</u>	<u>1989</u>	<u>1990</u>	<u>1991</u>
Common Loon	55	14	19	285	90
Pied-billed Grebe	56	30	5	14	9
Horned Grebe	27	6	4	12	6
Northern Gannett	4	8	35	2	9
American White Pelican	1	1	0	0	2
Brown Pelican	216	95	111	117	143
Double-crested Cormorant	173	117	235	110	132
Anhinga	0	0	0	1	2
American Bittern	0	0	2	2	0
Great Blue Heron	173	122	48	91	63
Great Egret	304	113	53	215	72
Snowy Egret	76	54	46	73	55
Little Blue Heron	16	13	6	9	5
Tri-colored Heron	19	16	11	13	16
Reddish Egret	1	2	2	6	6
Cattle Egret	5	0	0	3	1
Green-backed Heron	0	0	0	1	0
Black-crowned Night Heron	3	0	1	21	2
White Ibis	21	0	0	0	0
Snow Goose (blue form)	0	1	0	0	0
Snow Goose (white form)	0	4	0	0	0
Green-winged Teal	0	0	1	1	0
American Black Duck	2	0	6	0	0
Wood Duck	0	0	0	0	4
Mallard	1	0	1	1	0
Northern Shoveler	3	0	0	5	0
American Widgeon	0	0	0	1	0
Gadwall	8	0	0	0	0
Redhead	0	22	0	1	0
Ring-necked Duck	0	0	0	4	0
Greater Scaup	0	0	0	1	0
Lesser Scaup	1	36	0	4	1
scaup, spp.	17	1	430	19	0
Common Goldeneye	14	13	10	44	14
Bufflehead	38	?	16	143	38
Hooded Merganser	20	11	12	35	122
Red-breasted Merganser	435	149	124	390	167
Black vulture	7	3	0	0	1
Turkey vulture	75	30	28	87	20
Osprey	1	0	0	1	0
Bald Eagle*	3	2	2	6	5
Northern Harrier	6	16	11	2	2
Sharp-shinned Hawk	1	2	1	1	1
Cooper's Hawk	2	0	0	1	0
Red-shouldered Hawk	7	8	8	17	13
Red-tailed Hawk	11	13	12	7	5
American Kestrel	16	21	16	16	13
Merlin	0	0	0	1	1
Peregrine Falcon*	0	0	0	1	1
Northern Bobwhite	15	2	23	0	2
Clapper Rail	4	2	4	20	15
Sora	0	0	1	1	0
Virginia Rail	1	0	0	0	0
Common Moorhen	3	2	2	0	0
American Coot	18	6	4	3	0
Black-bellied Plover	50	56	83	144	114
Snowy Plover*	5	13	0	3	2

Appendix 1 cont'd.

	1987	1988	1989	1990	1991
Semipalmated Plover	40	56	32	36	106
Piping Plover*	1	2	7	6	31
Killdeer	33	48	12	200	14
American Oystercatcher	1	3	7	7	8
Greater Yellowlegs	4	3	9	34	10
Lesser Yellowlegs	1	6	0	5	2
Willet	256	537	315	124	300
Spotted Sandpiper	2	1	2	1	3
Whimbrel	0	6	0	0	5
Long-billed Curlew	0	0	0	0	1
Marbled Godwit	0	2	2	0	1
Ruddy Turnstone	20	46	46	69	71
Red Knot	4	60	30	266	54
Sanderling	33	128	44	53	71
Western Sandpiper	10	62	80	100	21
Least Sandpiper	62	7	14	24	30
Dunlin	279	551	574	377	702
peep spp.	0	40	45	190	0
Short-billed Dowitcher	84	109	144	74	277
Common Snipe	0	1	1	18	1
American Woodcock	2	0	5	1	0
Laughing Gull	146	149	556	425	273
Bonaparte's Gull	37	200	47	170	32
Ring-billed Gull	142	173	241	400	387
Herring Gull	74	120	109	60	36
Great Black-backed Gull	0	0	0	0	1
Caspian Tern	6	2	3	3	1
Royal Tern	70	118	53	24	133
Sandwich Tern	0	8	0	0	0
Forster's Tern	66	50	219	64	167
Black Skimmer	30	57	100	102	0
Rock Dove	29	15	11	10	26
Mourning Dove	18	9	2	8	1
Common Ground Dove	30	34	7	38	29
Eastern Screech Owl	3	1	4	11	6
Great Horned Owl	0	0	0	4	3
Barred Owl	0	2	2	3	2
Whip-poor-will	0	0	1	0	1
Belted Kingfisher	36	26	19	28	19
Red-bellied Woodpecker	14	17	16	56	33
Yellow-bellied Sapsucker	6	4	2	14	11
Downy Woodpecker	4	6	6	11	8
Red-cockaded Woodpecker*	0	0	3	0	0
Hairy Woodpecker	0	0	0	1	0
Northern Flicker	17	5	3	19	14
Pileated Woodpecker	0	0	2	3	2
Eastern Phoebe	14	16	16	26	18
Tree Swallow	772	155	75	120	130
Blue Jay	13	10	18	51	19
American Crow	6	13	135	36	36
Fish Crow	105	58	125	190	452
Carolina Chickadee	10	31	29	19	26
Tufted Titmouse	0	2	0	2	0
Redbreasted Nuthatch	0	0	0	7	3
Brownheaded Nuthatch	33	24	19	28	28
Carolina Wren	22	18	45	108	27
House Wren	26	18	23	40	32
Winter Wren	3	0	1	0	1
Sedge Wren	8	8	4	6	9

Appendix 1 cont'd.

	<u>1987</u>	<u>1988</u>	<u>1989</u>	<u>1990</u>	<u>1991</u>
Marsh Wren	10	4	6	21	30
Golden-crowned Kinglet	2	0	2	1	2
Ruby-crowned Kinglet	33	43	48	62	47
Blue-gray Gnatcatcher	6	2	4	0	2
Eastern Bluebird	17	32	38	36	67
Hermit Thrush	1	2	2	27	14
American Robin	757	8500	87	3300	3810
Gray Catbird	12	21	27	91	63
Northern Mockingbird	35	80	86	190	74
Brown Thrasher	5	2	3	9	0
Cedar Waxwing	0	15	0	1000	20
Water Pipit	0	0	1	0	0
Loggerhead Shrike	1	0	1	1	0
European Starling	130	209	348	103	58
White-eyed Vireo	1	1	6	4	1
Solitary Vireo	1	4	1	5	2
Orange-crowned Warbler	2	1	0	2	3
Yellow-rumped Warbler	487	780	633	2100	2205
Yellow-throated Warbler	4	0	0	1	0
Pine Warbler	44	23	55	43	26
Palm Warbler	0	6	6	20	20
Common Yellowthroat	30	18	24	58	37
Northern Cardinal	38	36	52	51	41
Rufous-sided Towhee	33	63	87	163	73
Chipping Sparrow	23	23	5	78	14
Field Sparrow	6	21	3	4	19
Vesper Sparrow	10	0	2	9	0
Savannah Sparrow	8	34	32	63	18
Grasshopper Sparrow	0	0	0	1	0
Sharp-tailed Sparrow	4	3	5	13	8
Seaside Sparrow	0	3	0	3	1
Song Sparrow	11	28	24	105	34
Swamp Sparrow	27	10	31	58	28
White-throated Sparrow	1	6	10	44	20
Dark-eyed Junco	0	1	0	0	0
Red-winged Blackbird	684	129	697	140	133
Eastern Meadowlark	31	52	29	81	34
Brewer's Blackbird	3	0	0	0	0
Boat-tailed Grackle	1	0	0	0	0
Common Grackle	0	25	100	63	216
Brown-headed Cowbird	0	0	1	90	1
House Finch	0	0	0	0	1
Pine Siskin	0	40	0	15	0
American Goldfinch	20	15	12	61	13
House Sparrow	21	12	19	17	4
<hr/>					
<i>Number of Species</i>	<i>121</i>	<i>117</i>	<i>119</i>	<i>134</i>	<i>123</i>
<i>Total No. of Individuals</i>	<i>6,986</i>	<i>14,486</i>	<i>7,126</i>	<i>13,743</i>	<i>11,972</i>

*Endangered, Threatened or Candidate Species under the Federal Endangered Species Act.

APPENDIX TWO

THREATENED AND

ENDANGERED

SPECIES



APPENDIX 2

THREATENED, ENDANGERED, AND OTHER SPECIES OF CONCERN LIKELY TO OCCUR IN GULF COUNTY, FLORIDA Compiled by U.S. Fish and Wildlife Service, June 2000

Common Name	Scientific Name	Status State	Status FWS	Natural Communities
FISH:				
Gulf sturgeon	<i>Acipenser oxyrinchus desotoi</i>	SSC	T	ESTUARINE: various MARINE: various habitats RIVERINE: alluvial and blackwater streams
Bluenose shiner	<i>Pteronotropsis welaka</i>	SSC		RIVERINE: blackwater, alluvial, and spring-run streams
AMPHIBIANS & REPTILES:				
American alligator	<i>Alligator mississippiensis</i>	SSC	T(s/a)	ESTUARINE: marshes, various habitats LACUSTRINE: marshes, swamps, various habitats PALUSTRINE: swamps, floodplains, marshes, various habitats RIVERINE: open water, shorelines, various habitats
Flatwoods salamander	<i>Ambystoma cingulatum</i>		T	PALUSTRINE: wet flatwoods, dome swamp, basin swamp, ruderal TERRESTRIAL: mesic flatwoods (reproduces in ephemeral wetlands within this community)
Loggerhead turtle	<i>Caretta caretta</i>	T	T	TERRESTRIAL: sandy beaches; nesting
Green turtle	<i>Chelonia mydas mydas</i>	E	E	TERRESTRIAL: sandy beaches; nesting
Leatherback turtle	<i>Dermochelys coriacea</i>	E	E	TERRESTRIAL: sandy beaches; nesting
Eastern indigo snake	<i>Drymarchon corais couperi</i>	T	T	ESTUARINE: tidal swamp PALUSTRINE: hydric hammock, wet flatwoods TERRESTRIAL: mesic flatwoods, upland pine forest, sandhills, scrub, scrubby flatwoods, rockland hammock, ruderal
Hawksbill turtle	<i>Eretmochelys imbricata imbricata</i>	E	E	MARINE: open water; no nesting
Coat skink	<i>Eumeces anthracinus</i>		ce	PALUSTRINE: seepage slope, baygall TERRESTRIAL: upland pine forest, upland hardwood forest, mesic flatwoods
Gopher tortoise	<i>Gopherus polyphemus</i>	SSC	ce	TERRESTRIAL: sandhills, scrub, scrubby flatwoods, xeric hammocks, coastal strand, ruderal
Barbour's map turtle	<i>Graptemys barbouri</i>	SSC	ce	PALUSTRINE: floodplain stream, floodplain swamp RIVERINE: alluvial stream

E = endangered, T = threatened, P = proposed, C = candidate, s/a = Similarity of appearance,
SSC = Species of Special Concern, ce = Consideration encouraged, CH = Critical habitat

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Common Name	Scientific Name	Status State	Status FWS	Natural Communities
Alligator snapping turtle	<i>Macrolemys temminckii</i>	SSC	ce	ESTUARINE: tidal marsh LACUSTRINE: river floodplain lake, swamp lake RIVERINE: alluvial stream, blackwater stream
Gulf salt marsh snake	<i>Nerodia clarkii clarkii</i>		ce	ESTUARINE: tidal marsh, tidal swamp MARINE: tidal marsh, tidal swamp
Florida pine snake	<i>Pituophis melanoleucus mugitus</i>	SSC	ce	LACUSTRINE: ruderal, sandhill upland lake TERRESTRIAL: sandhill, scrubby flatwoods, xeric hammock, ruderal
Suwannee cooter	<i>Pseudemys concinna suwanniensis</i>	SSC		RIVERINE: alluvial stream, blackwater stream, spring-fed stream
Gopher frog	<i>Rana capito</i>	SSC	ce	TERRESTRIAL: sandhill, scrub, scrubby flatwoods, xeric hammock (reproduces in ephemeral wetlands within these communities)
BIRDS:				
Bachman's sparrow	<i>Aimophila aestivalis</i>		ce	TERRESTRIAL: various, ruderal
Limpkin	<i>Aramus guarauna</i>	SSC		LACUSTRINE: various PALUSTRINE: various RIVERINE: various
Southeastern snowy plover	<i>Charadrius alexandrinus tenuirostris</i>	T	ce	ESTUARINE: exposed unconsolidated substrate MARINE: exposed unconsolidated substrate TERRESTRIAL: dunes, sandy beaches, and inlet areas
Piping plover	<i>Charadrius melodus</i>	T	T	ESTUARINE: exposed unconsolidated substrate MARINE: exposed unconsolidated substrate TERRESTRIAL: dunes, sandy beaches, and inlet areas. Mostly wintering and migrants.
Little blue heron	<i>Egretta caerulea</i>	SSC		ESTUARINE: marshes, shoreline PALUSTRINE: floodplains, swamps RIVERINE: shoreline
Snowy egret	<i>Egretta thula</i>	SSC		ESTUARINE: marshes, tidal swamps, shoreline LACUSTRINE: lake edges PALUSTRINE: swamp, floodplain, ruderal RIVERINE: shoreline

E = endangered, T = threatened, P = proposed, C = candidate, s/a = Similarity of appearance,
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Common Name	Scientific Name	Status State	Status FWS	Natural Communities
Tricolored heron	<i>Egretta tricolor</i>	SSC		ESTUARINE: marshes, tidal swamps, shoreline LACUSTRINE: lake edges PALUSTRINE: swamp, floodplain, ruderal RIVERINE: shoreline
Arctic peregrine falcon	<i>Falco peregrinus tundrius</i>	E	E(s/a)	ESTUARINE: winters along coasts LACUSTRINE: various PALUSTRINE: various TERRESTRIAL: various, ruderal
Southeastern kestrel	<i>Falco sparverius paulus</i>	T	ce	ESTUARINE: various habitats PALUSTRINE: various habitats TERRESTRIAL: open pine forests, clearings, ruderal, various
Florida sandhill crane	<i>Grus canadensis pratensis</i>	T		LACUSTRINE: flatwoods/prairie lake, marsh lake PALUSTRINE: swale, basin marsh, depression marsh, bog, marl prairie, wet prairie TERRESTRIAL: dry prairie, ruderal
American oystercatcher	<i>Haematopus palliatus</i>	SSC		ESTUARINE: exposed unconsolidated substrate, exposed mollusk reef MARINE: exposed unconsolidated substrate, exposed mollusk reef TERRESTRIAL: beaches, ruderal areas
Bald eagle	<i>Haliaeetus leucocephalus</i>	T	T	ESTUARINE: marsh edges, tidal swamp, open water LACUSTRINE: swamp lakes, edges PALUSTRINE: swamp, floodplain RIVERINE: shoreline, open water TERRESTRIAL: pine and hardwood forests, clearings
Wood stork	<i>Mycteria americana</i>	E	E	ESTUARINE: marshes LACUSTRINE: floodplain lakes, marshes (feeding), various PALUSTRINE: marshes, swamps, various
Brown pelican	<i>Pelecanus occidentalis</i>	SSC		ESTUARINE: islands for nesting, open water MARINE: open water
Red-cockaded woodpecker	<i>Picoides borealis</i>	T	E	TERRESTRIAL: mature pine forests

E = endangered, T = threatened, P = proposed, C = candidate, s/a = Similarity of appearance,
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THREATENED, ENDANGERED, AND OTHER SPECIES OF CONCERN LIKELY TO OCCUR IN GULF COUNTY, FLORIDA
Compiled by U.S. Fish and Wildlife Service, June 2000

Common Name	Scientific Name	Status State	Status FWS	Natural Communities
Least tern	<i>Sterna antillarum</i>	T		ESTUARINE: various LACUSTRINE: various RIVERINE: various TERRESTRIAL: beach dune, ruderal. Nests common on rooftops.
MAMMALS:				
St. Andrew beach mouse	<i>Peromyscus polionotus peninsularis</i>	E	E	TERRESTRIAL: beach dune, coastal scrub. Bay Co. sites: Tyndall AFB (Crooked Beach canal), Gulf Co. mainland east to Mexico Beach canal), Gulf Co. sites: St. Joe Beach, St. Joseph Peninsula State Park, St. Joseph Peninsula, Cape San Blas (includes Eglin AFB), Cape San Blas east to Money Bayou.
Southeastern big-eared bat	<i>Plecotus rafinesquii</i>		ce	PALUSTRINE: various, floodplains TERRESTRIAL: pine and hardwood forests, ruderal, various
West Indian manatee	<i>Trichechus manatus latirostris</i>	E	E	ESTUARINE: submerged vegetation, open water MARINE: open water, submerged vegetation RIVERINE: alluvial stream, blackwater stream, spring-run stream
Florida black bear	<i>Ursus americanus floridanus</i>	T	ce	PALUSTRINE: titi swamps, floodplains TERRESTRIAL: pine and hardwood forests
INVERTEBRATES:				
Fat three-ridge	<i>Amblema neislerii</i>		E	RIVERINE: main channels of small to large rivers; slow to moderate currents; various substrates (Panhandle watersheds: Apalachicola, Chipola)
Purple bankclimber	<i>Elliptioideus sloatianus</i>		T	RIVERINE: small to large rivers in slow to moderate currents over sand, sand mixed with mud, or gravel substrates (Panhandle watersheds: Apalachicola, Ochlockonee)
PLANTS:				

E = endangered, T = threatened, P = proposed, C = candidate, s/a = Similarity of appearance,
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Common Name	Scientific Name	Status State	Status FWS	Natural Communities
Southern milkweed	<i>Asclepias viridula</i>	T	ce	PALUSTRINE: wet prairie, seepage slope edges RIVERINE: seepage stream banks TERRESTRIAL: mesic flatwoods, drainage ditches
Pine-woods aster	<i>Aster spinulosus</i>	E	ce	PALUSTRINE: seepage slope TERRESTRIAL: sandhill, scrubby and mesic flatwoods
Toothed savory	<i>Calamintha dentata</i>		ce	TERRESTRIAL: sandhill, roadsides
Tropical waxweed	<i>Cuphea aspera</i>		ce	PALUSTRINE: wet prairie, seepage slope TERRESTRIAL: mesic flatwoods
Spoon-leaved sundew	<i>Drosera intermedia</i>	T		LACUSTRINE: sinkhole lake edges PALUSTRINE: seepage slope, wet flatwoods, depression marsh RIVERINE: seepage stream banks, drainage ditches
Telephus spurge	<i>Euphorbia telephioides</i>	E	T	TERRESTRIAL: mesic flatwoods; disturbed wiregrass (<i>Aristida stricta</i>) areas, coastal scrub. All known sites are within 4 miles of Gulf of Mexico.
Wiregrass gentian	<i>Gentiana pennelliana</i>	E	ce	PALUSTRINE: seepage slope, wet prairie, roadside ditches TERRESTRIAL: mesic flatwoods, planted slash pine
Panhandle spiderlily	<i>Hymenocallis henryae</i>	E	ce	PALUSTRINE: dome swamp edges, wet prairie, wet flatwoods, baygall edges, swamp edges TERRESTRIAL: wet prairies and flatwoods
Thick-leaved water willow	<i>Justicia crassifolia</i>	E	ce	PALUSTRINE: dome swamp, seepage slope TERRESTRIAL: mesic flatwoods
Southern red lily	<i>Lilium catesbaei</i>	T		PALUSTRINE: wet prairie, wet flatwoods, seepage slope TERRESTRIAL: mesic flatwoods, seepage slope; usually with grasses
Harper's grooved yellow flax	<i>Linum sulcatum var harperi</i>		ce	PALUSTRINE: wet flatwoods TERRESTRIAL: mesic flatwoods; in site-prepped areas

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Common Name	Scientific Name	Status State	Status FWS	Natural Communities
West's flax	<i>Linum westii</i>	E	ce	PALUSTRINE: dome swamp, depression marsh, wet flatwoods, wet prairie, pond margins
Gulf coast lupine	<i>Lupinus westianus</i>	T	ce	TERRESTRIAL: beach dune, scrub, disturbed areas, roadsides, blowouts in dunes
White birds-in-a-nest	<i>Macbridea alba</i>	E	T	PALUSTRINE: seepage slope TERRESTRIAL: grassy mesic pine flatwoods, savannahs, roadsides, and similar habitat.
Piedmont water-milfoil	<i>Myriophyllum laxum</i>		ce	LACUSTRINE: sandhill upland lake, submersed PALUSTRINE: floodplain and dome swamp RIVERINE: blackwater stream, roadside ditches
Giant water-dropwort	<i>Oxypolis filiformis greenmanii</i>	E		PALUSTRINE: dome swamp, wet flatwoods, ditches; in water
Godfrey's (violet) butterwort	<i>Pinguicula ionantha</i>	E	T	PALUSTRINE: wet flatwoods, wet prairie, bog; in shallow water RIVERINE: seepage slope; in shallow water. Also, roadside ditches and similar habitat.
Yellow butterwort	<i>Pinguicula lutea</i>	T		PALUSTRINE: flatwoods, bogs
Chapman's butterwort	<i>Pinguicula planifolia</i>	T	ce	PALUSTRINE: wet flatwoods, seepage slopes, bog, dome swamp, ditches; in water
Bent golden aster	<i>Pityopsis flexuosa</i>	E	ce	TERRESTRIAL: sandhill, upland pine forest, ruderal
Yellow fringed orchid	<i>Platanthera ciliaris</i>	T		PALUSTRINE: bogs, wet flatwoods TERRESTRIAL: Bluff
Yellow fringeless orchid	<i>Platanthera integra</i>	E	ce	PALUSTRINE: wet prairie, seepage slope TERRESTRIAL: mesic flatwoods
Snowy orchid	<i>Platanthera nivea</i>	T		PALUSTRINE: bogs
Meadowbeauty	<i>Rhexia parviflora</i>	E	ce	PALUSTRINE: dome swamp margin, seepage slope, depression marsh; on slopes; with hypericum

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Common Name	Scientific Name	Status State	Status FWS	Natural Communities
Chapman's rhododendron	<i>Rhododendron chapmanii</i>	E	E	PALUSTRINE: seepage slope (titi bog) TERRESTRIAL: mesic flatwoods; ecotone between flatwoods or more xeric longleaf communities and titi bogs.
White-top pitcher plant	<i>Sarracenia leucophylla</i>	E	ce	PALUSTRINE: wet prairie, seepage slope, baygall edges, ditches
Hooded pitcher plant	<i>Sarracenia minor</i>	T		PALUSTRINE: wet flatwoods, wet prairie, seepage slope
Parrot pitcher plant	<i>Sarracenia psittacina</i>	T		PALUSTRINE: wet flatwoods, wet prairie, seepage slope
Decumbant pitcher plant	<i>Sarracenia purpurea</i>	T		PALUSTRINE: Bogs
Florida skullcap	<i>Scutellaria floridana</i>	E	T	PALUSTRINE: seepage slope, wet flatwoods, grassy openings TERRESTRIAL: mesic flatwoods
Chapman's crownbeard	<i>Verbesina chapmanii</i>	T	ce	PALUSTRINE: seepage slope TERRESTRIAL: mesic flatwoods with wiregrass (<i>Aristida stricta</i>)
Drummond's yellow-eyed grass	<i>Xyris drummondii</i>		ce	PALUSTRINE: wet flatwoods, bog, seepage slopes, ditches
Quillwort yellow-eyed grass	<i>Xyris isoetifolia</i>		ce	LACUSTRINE: sandhill upland lake margins
Harper's yellow-eyed grass	<i>Xyris scabrifolia</i>	T	ce	PALUSTRINE: wet flatwoods, wet prairie
				PALUSTRINE: seepage slope, wet prairie, bogs

E = endangered, T = threatened, P = proposed, C = candidate, s/a = Similarity of appearance,
SSC = Species of Special Concern, ce = Consideration encouraged, CH = Critical habitat

APPENDIX THREE

SEDIMENT SAMPLING AND ANALYSES

**STANDARD OPERATING PROCEDURES
FOR SEDIMENT SAMPLING**

**ANALYTICAL METHODS
UNIVERSITY OF MISSOURI**

**ANALYTICAL METHODS
MISSISSIPPI STATE CHEMICAL
LABORATORY**

**ORGANIC CARBON DETERMINATION
UNIVERSITY OF SOUTH ALABAMA**

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STANDARD OPERATING PROCEDURES

SEDIMENT SAMPLING FOR CHEMICAL ANALYSES

To maintain and assure quality control, sediment samples collected for shipment to USFWS-approved analytical laboratories will be obtained and handled as follows:

COLLECTION OF SAMPLES FROM COASTAL WATERS OR LARGE RIVERS

1. **Sampling Devices** - The following devices are approved for obtaining sediment samples:
 - a) Ponar grab, Standard. Manufactured from 316 stainless steel including jaws, side plates, underlip plate, screen frame, screens and hinge pin. 583 micron mesh top screens; weight empty - 21 kg (45 lbs); sampling area 22.85 cm x 22.85 cm (9" x 9").
 - b) Ponar grab, Petite. Manufactured with 316 stainless steel including jaws, side plates, underlip plate, screen frame, screens and hinge pin. 583 micron mesh top screens; weight empty - 6.8 kg (15 lbs); sampling area 15.24 x 15.24 cm (6" x 6").
2. **Sediment Sampling Boat** -
 - A) fiberglass boat with outboard motor equipped as follows:
 - 1) navigation and positioning capabilities including: a) loran navigation system, b) chart-printing depth recorder, c) compass, d) appropriate navigation charts.
 - 2) 12 volt electric winch; steel ginpole with heavy duty pulley; 100' of 1/2" braided nylon lift rope.
 - 3) stainless steel wash deck equipped with an overboard wash pump.

3. Other Equipment and Supplies -

- A) Stainless steel sample pan 28 x 48 x 10 cm.
- B) Pre-cleaned, chemical-free, glass 1.0 liter sample jars with screw-top lids having teflon liners.
- C) Pre-cleaned, chemical-free stainless steel utensils.
- D) Clean insulated ice chests with ice.
- E) Permanent, glass-adhesive markers.
- F) Bound collection log-book or individual record sheets.
- G) Disposable laboratory gloves.
- F) Meters: dissolved oxygen, salinity, temperature, pH and others, as appropriate.

4. Operational Procedures -

- 1) Prior to each *collection day* the ponar sampler will be scrubbed and washed with a detergent solution, rinsed thoroughly with tap water, and then rinsed with distilled water. After each collection *field trip* the ponar will be cleaned, as above, and stored properly.
- 2) The daily collection plan shall provide, to the greatest extent possible, for sampling to begin at the least contaminated station, with work advancing toward the most contaminated station.
- 3) Sediment samples obtained at *sampling stations* will be composite samples. Each composite will consist of five individual ponar sub-samples collected 3 meters apart along a straight-line transect, with the collection boat anchored. Move from one *sub-sample position* to the next by slipping the anchor line to provide approximately 3 meters of horizontal drift.
- 4) Place each ponar sub-sample in the sample pan. Take approximately 150 grams of sediment from the center of the sub-sample using appropriate utensils and place it in the collection jar designated for that station. After obtaining each sub-sample, rinse utensils, wash deck, sample pan, and the ponar sampler with seawater or river water.

Note: 150 grams of sub-sample collected from each of the 5 sub-sample positions (about 750 grams of sample total) should result in the sample jar being about 3/4 full. This leaves adequate space in the jar for any expansion of the sample during freezing.

- 5) During collection of the third ponar sub-sample, record the *station location* by loran positions and by latitude and longitude. At this time, also record all other station information (such as depth, salinity, water temperature, etc).
- 6) Place each sub-sample (total, n=5) in the appropriate pre-labeled, sample jar. Secure the lid and place sample on ice in a cooler.
- 7) After work at each *sampling station* is complete, clean the ponar, sample pan, wash deck and utensils thoroughly and rinse with seawater or river water.
- 8) For field trips involving more than one day, samples will be frozen and stored in a portable *field freezer*.
- 9) After each collection day, double-wrap each full sample jar with clean, heavy-duty aluminum foil, place a second identification label over the foil, and store in a freezer.
- 10) Upon returning to the Panama City Field Office samples will be transferred to a *laboratory freezer* and held at -23° degrees centigrade (-10 fahrenheit) until shipment for chemical analyses. Sediment samples for particle size analysis will be held at 4° degrees C.



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% MOISTURE

For animal tissue and sediments of sufficient size, moisture was determined by placing a weighed aliquot of the sample in a Fisher Isotemp oven and drying at 103-105°C. The dried sample was then weighed and the data entered into a computer program to generate the % moisture and final report.

Plants, and samples too small for oven dried moisture determination had the % moisture calculated from the moisture lost during the freeze-drying in the Labcono Freeze-Dryer 8. The data was entered into a computer program to generate a % moisture and final report.

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HOMOGENIZATION

Large tissue samples, such as whole fish, were first run through a meat grinder one or more times depending on the size of the sample. An aliquot of the ground sample was weighed and frozen. For smaller tissue samples and plant samples the entire sample was weighed and then frozen. For sediments, the sample was mixed and an aliquot weighed and frozen. The frozen samples were placed in a Labcono Freeze Dryer 8 until the moisture had been removed. The dry samples were then weighed and further homogenized using a blender, or Spex Industries, Inc. Model 8000 mixer/mill with tungsten-carbide vial and balls.

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NITRIC - PERCHLORIC DIGESTION - (SELENIUM)

Approximately 0.5 g. of sample was weighed into a freshly cleaned 100 ml. quartz Kjeldahl flask. (Sediment samples and samples containing a high percent of silica were digested in 100 ml. teflon breakers.) For water samples, 50 ml. of sample were measured into a teflon beaker. Slowly 15 ml. of concentrated sub-boiled HNO_3 and 2.5 ml. of concentrated sub-boiled HClO_4 were added. Foaming may occur with some samples. If the foaming started to become excessive, the container was cooled in a beaker of cold water. After the initial reaction had subsided, the sample was placed on low heat until the evolution of dark red fumes had ceased. Gradually, the heat was increased until the HNO_3 began refluxing, samples were allowed to reflux overnight. (This decreased the chance for charring during the reaction with HClO_4 .) After the refluxing, the heat was gradually increased until the HNO_3 had been driven off, and the reaction with HClO_4 had occurred. When dense white fumes from the HClO_4 were evident, the samples were removed from the heat and allowed to cool. Two ml. of concentrated sub-boiled HCl were added. The flasks were replaced on the heat and warmed until the containers were hot to the touch or started to boil. They were removed from the heat, and 5-10 ml. of deionized water were added. Samples were allowed to cool. They were then diluted using deionized water in a 50 ml. volumetric flask and transferred to clean, labeled, 2 oz. polyethylene bottles.

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NITRIC - PERCHLORIC DIGESTION - (ARSENIC)

Approximately 0.5 g. of sample was weighed into a freshly cleaned 100 ml. Kjeldahl flask. (Sediment samples and samples containing a high percent of silica were digested in 100 ml. teflon beakers.) For water samples, 50 ml. of sample were measured into a teflon beaker. Slowly 15 ml. of concentrated sub-boiled HNO_3 and 2.5 ml. of concentrated sub-boiled HClO_4 were added. Foaming may occur with some samples. If the foaming started to become excessive, the container was cooled in a beaker of cold water. After the initial reaction had subsided, the sample was placed on low heat until the evolution of dark red fumes had ceased. Gradually, the heat was increased until the HNO_3 had been driven off, and the reaction with HClO_4 had occurred. After this reaction, the samples were heated approximately 5 minutes, after dense white fumes from the HClO_4 were evident. The samples were removed from the heat and allowed to cool. Samples were diluted using deionized water in 50 ml. volumetric flasks and transferred to clean, labeled, 2 oz. polyethylene bottles.

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NITRIC - PERCHLORIC DIGESTION - (ICP)

Approximately 0.5 g. of sample was weighed into a freshly cleaned 100 ml. quartz Kjeldahl flask. (Sediment samples and samples containing a high percent of silica were digested in 100 ml. teflon beakers.) For water samples, 50 ml. of sample were measured into a teflon beaker. Slowly 15 ml. of concentrated sub-boiled HNO_3 and 2.5 ml. of concentrated sub-boiled HClO_4 were added. Foaming may occur with some samples. If the foaming started to become excessive, the container was cooled in a beaker of cold water. After the initial reaction had subsided, the sample was placed on low heat until the evolution of dark red fumes had ceased. Gradually, the heat was increased until the HNO_3 began refluxing, samples were allowed to reflux overnight. (This decreased the chance for charring during the reaction with HClO_4 .) After the refluxing, the heat was gradually increased until the HNO_3 had been driven off, and the reaction with HClO_4 had occurred. When dense white fumes from the HClO_4 were evident, the samples were removed from the heat and allowed to cool. Two ml. of concentrated sub-boiled HCl were added. The flasks were replaced on the heat and warmed until the containers were hot to the touch or started to boil. They were removed from the heat, and 5-10 ml. of deionized water were added. Samples were allowed to cool. They were then diluted using deionized water in a 50 ml. volumetric flask and transferred to clean, labeled, 2 oz. polyethylene bottles.

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NITRIC REFLUX DIGESTION FOR MERCURY

Approximately 0.5 g. of sample was weighed into a freshly cleaned 50 ml. round bottom flask with 24/40 ground glass neck. For waters, 10 ml. of sample were measured into the flask. Five ml. of concentrated sub-boiled HNO_3 were added and the flask was placed under a 12 inch water-cooled condenser with water running through the condenser. The heat was turned up to allow the HNO_3 to reflux no more than 1/3 the height of the columns. Samples were allowed to reflux for two hours. Then the heat was turned off and the samples allowed to cool. The condensers were rinsed with 1% v/v HCl and the flasks removed. The samples were diluted with 1% v/v HCl in a 50 ml. volumetric flask and then transferred to clean, labeled, 2 oz. flint glass bottles.

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ARSENIC AND SELENIUM BY HYDRIDE

The Varian VGA-76 hydride generation accessory was mounted on either a Perkin-Elmer Model 603 AA or Model 3030 (B) AA. Electrodeless Discharge lamps (EDL) were used. The instrument and EDL settings were taken from the instrument manuals. The burner mount for a Perkin-Elmer Model 10 Hydride generator was modified slightly to hold the Varian quartz cell. The cell was aligned in the light path of the burner chamber and a very lean flame was used for heating the cell. The two stock solutions were 50% v/v sub-boiled HCl and 0.6% NaBH₄ in 0.5% NaOH for Selenium and concentrated sub-boiled HCl and 1% NaBH₄ in 0.5% NaOH for Arsenic. Samples were diluted with 10% v/v sub-boiled HCl. Standards were prepared by dilution of Fisher 1000 ppm stock with 10% v/v sub-boiled HCl in the range of 0 to 20 PPB. The instrument was standardized to read directly in PPB using S1 = 5.00 and S2 = 20.00. After standardization, the standardization was checked by reading other standards such as 2.00, 10.00 and 15.00 PPB and an instrumental quality control sample with a known value. If the standards and quality control were acceptable, the detection limit was determined by reading the zero standard 10 times, and twice the standard deviation of the mean was used as the detection limit. Samples were analyzed by taking an integrated reading for 3 seconds after the plateau was reached for the sample. This occurred approximately 45 seconds after the sample tube was placed in the sample. Standardization was checked every 8-15 samples and approximately 10% of the samples were checked by the method of additions to monitor matrix effects. Matrix effects were usually not significant with the VGA-76. The data was corrected for drift of the standard curve and entered into the AA calculation program. This program corrected for blank, dilution, sample weight, sample volume and recorded the data in the LIMS database for report generation.

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MERCURY - COLD VAPOR ATOMIC ABSORPTION

Equipment used for Cold Vapor Atomic Absorption include: Perkin-Elmer Model 403 AA; Perkin-Elmer Model 056 recorder; Technicon Sampler I; Technicon Pump II; a glass cell with quartz windows and capillary tube for entry and exit of the mercury vapor; and a liquid-gas separator. The samples were placed in 4 ml. sample cups at least 3/4 full. The samples were mixed with hydroxylamine for preliminary reduction, then stannous chloride for reduction to the mercury vapor. The vapor was separated from the liquid and passed through the cell mounted in the light path of the burner compartment. The peaks were recorded and the peak heights measured. The standardization was done with at least 5 standards in the range of 0 to 10 ppb. The correlation coefficient was usually 0.9999 or better and must have been at least 0.999 to have been acceptable. A standard was run every 8-10 samples to check for drift in the standardization. This was usually less than 5%. Standards were preserved with 10% v/v HNO_3 , 1% v/v HCl and 0.05% w/v $\text{K}_2\text{Cr}_2\text{O}_7$. The solution concentrations were calculated and the data entered into the AA calculation program which corrected for blank, dilution, sample weight, sample volume and entered the data into the LIMS system for report generation.

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INDUCTIVELY COUPLED PLASMA (ICP)

The instrument used for ICP analysis was a Jarrell-Ash Model 1100, Mark III with 40 analytical channels, controlled by a Digital Equipment Company (DEC) 11/23+ computer with two RL02 disk drives, DEC VT100 terminal, and DEC LA120 decwriter III. The instrument was standardized with a series of seven standards containing 36 elements. After the standardization, the detection limit was determined by taking ten integrations of the zero standard; three times the standard deviation of the mean was used as the detection limit. Instrumental quality control samples were then analyzed to check the ICP operation. If the values were acceptable, the samples were then analyzed. Standards were run every 10-15 samples to check for drift. If the drift was more than 5%, the instrument was restandardized. After the analyses were completed, the data were transferred to the Perkin-Elmer LIMS 2000 computer for calculation. The final detection limit for each element was further increased by 4% of the magnitude of the spectral interferences from the other elements. The data were checked before calculation to correct for possible errors in sample number, weight, volumes and dilution. The data were calculated using the ICP calculation program written by ETSRC computer staff, which corrected for blanks, standard drift, spectral interferences, sample weight, sample volume, and dilution. After the quality control was reviewed, a final report was generated using a Hewlett-Packard laser jet printer.

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NITRIC DIGESTION FOR GRAPHITE FURNACE

Approximately 0.5 g. of sample was weighed into a freshly cleaned 100 ml. Kjeldahl flask. (Sediment samples and samples containing a high percent of silica were digested in 100 ml. teflon beakers.) For water samples, 50 ml. of sample were measured into a teflon beaker. Slowly, 15 ml. of sub-boiled HNO_3 were added. Foaming may occur with some samples. If the foaming started to become excessive, the flask was cooled in a beaker of cold water. The samples were placed on low heat until the initial reaction and evolution of dark red fumes had ceased. Gradually, the heat was increased until the volume was reduced to approximately 2 ml. The sample was removed from the heat and allowed to cool. Samples were diluted using deionized water in a 50 ml. volumetric flask and transferred to clean, labeled, 2 oz. polyethylene bottles.

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GRAPHITE FURNACE AA

The instruments used for graphite furnace AA, were either a Perkin-Elmer Model 3030B with Model HGA-500 graphite furnace, Model AS-40 autosampler and Model 056 recorder, or the Perkin-Elmer Model 5100 Zeeman with Model HGA-600 graphite furnace, Model AS-60 autosampler and Model 7300 computer. The conditions for a particular element were set up according to the instruction manual. The L'vov platform and appropriate matrix modifier were used. A standard curve and known quality control sample were run to check the instrument operation. The method of standard additions was used on a minimum of 1 out of 5 samples. If the average slopes for the standard additions gave a %RSD of 5% or less then the average slope was used to calculate the sample concentrations. If the average slope was not acceptable then the samples all had to be run using the method of standard additions. After calculating the solution concentrations, the data were entered into a computer program that corrected for blank, dilution, sample weight and volume, and entered the data into the LIMS data base for report generation.

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Analytical Methods
Mississippi State Chemical Laboratory
Mississippi State University
Box CR
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Method 1. Analysis For Organochlorine Pesticides and PCBs In Animal and Plant Tissue.

Ten gram tissue samples are thoroughly mixed with anhydrous sodium sulfate and soxhlet extracted with hexane for seven hours. The extract is concentrated by rotary evaporation; transferred to a tared test tube, and further concentrated to dryness for lipid determination. The weighed lipid sample is dissolved in petroleum ether and extracted four times with acetonitrile saturated with petroleum ether. Residues are partitioned into petroleum ether which is washed, concentrated, and transferred to a glass chromatographic column containing 20 grams of Florisil. The column is eluted with 200 ml 6% diethyl ether/94% petroleum ether (Fraction I) followed by 200 ml 15% diethyl ether/85% petroleum ether (Fraction II). Fraction II is concentrated to appropriate volume for quantification of residues by packed or capillary column electron capture gas chromatography. Fraction I is concentrated and transferred to a Silicic acid chromatographic column for additional cleanup required for separation of PCBs from other organochlorines. Three fractions are eluted from the silicic acid column. Each is concentrated to appropriate volume for quantification of residues by packed or megabore column, electron capture gas chromatography. PCBs are found in Fraction II.

Method 2. Analysis For Organochlorine Pesticides and PCBs In Soil and Sediment.

Twenty-five gram soil or sediment samples are extracted with acetone followed by hexane, by allowing to soak one hour in each with intermittent shaking. The combined extracts are centrifuged and decanted into a separatory funnel containing sufficient water to facilitate partitioning of residues into hexane portion. The hexane is washed twice with water and concentrated to appropriate volume for transfer to a 1.6 gram Florisil mini-column topped with 1.6 grams sodium sulfate. Residues are eluted from the column in two elution fractions. Fraction I consists of 12 milliliters hexane followed by 12 milliliters of 1% methanol in hexane, and Fraction II consists of an additional 24 milliliters of 1% methanol in hexane. If additional cleanup is required to separate PCBs from other organochlorines in Fraction I, further chromatography on a Silicic acid column is performed. Quantification of residues in the two Florisil fractions and three Silicic acid fractions is by packed or megabore column, electron capture gas chromatography.

Method 3. Analysis For Aliphatic and Polynuclear Aromatic Hydrocarbons In Animal and Plant Tissue.

A sample of appropriate size (i.e. 15 grams animal or plant tissue, 2 grams adipose, 5 grams eggs) is digested in 6N aqueous potassium hydroxide for 24 hours at 35 °C. Cool digestate thoroughly in an ice bath and carefully neutralize with glacial acetic acid. Extract the neutralized reaction mixture three times with methylene chloride; concentrate the combined extracts to near dryness and reconstitute in petroleum ether for transfer to a 20 gram 1% deactivated silica gel column, topped with 5 grams neutral alumina. Aliphatic and polynuclear aromatic hydrocarbon residues are separated by eluting aliphatics from the column with 100 ml petroleum ether (Fraction I) followed by elution of aromatics using first, 100ml 40% methylene chloride/60% petroleum ether, then 50 ml methylene chloride (Combined eluates, Fraction II). If needed, Fraction I containing aliphatics is subjected to additional cleanup by concentration and transfer to a deactivated (2% water) Florisil column. Aliphatic residues are eluted from the Florisil column using 200 ml 6% diethyl ether/94% petroleum ether. The eluate is concentrated to appropriate volume for quantification by capillary column, flame ionization gas chromatography. The silica gel Fraction II containing aromatic hydrocarbons is concentrated, reconstituted in methylene chloride, and subjected to gel permeation chromatography (GPC) cleanup prior to quantification by capillary, flame ionization gas chromatography and fluorescence HPLC.

Method 4. Analysis For Aliphatic and Aromatic Hydrocarbons In Soil and Sediment.

Twenty gram soil or sediment samples are extracted with acetone, followed by petroleum ether, by allowing to soak one hour in each with intermittent shaking. A final acetone/petroleum ether extraction is done, and the extracts are combined, centrifuged, and transferred to a separatory funnel containing sufficient water to facilitate partitioning of residues into petroleum ether portion. The petroleum ether is washed twice with water and concentrated by Kuderna-Danish to appropriate volume for transfer to a 20 gram 1% deactivated silica gel column, topped with five grams neutral alumina. Aliphatic and polynuclear aromatic hydrocarbon residues are fractionated by eluting aliphatics from the column with 100 ml petroleum ether (Fraction I) followed by elution of aromatics using first, 100 ml 40% methylene chloride/60% petroleum ether, then 50 ml methylene chloride (Combined eluates, Fraction II). If needed, Fraction I containing aliphatics is subjected to additional cleanup by concentration and transfer to a deactivated (2% water) Florisil column. Aliphatic residues are eluted from the Florisil column using 200 ml 6% diethyl ether/94% petroleum ether. The eluate is concentrated to appropriate volume for quantification by capillary column, flame ionization gas chromatography. The silica gel Fraction II containing aromatic hydrocarbons is concentrated, reconstituted in methylene chloride, and subjected to gel permeation chromatographic (GPC) cleanup prior to quantification by capillary, flame ionization gas chromatography and fluorescence HPLC.

Elution Profiles for Florisil, Silica Gel and
Silicic Acid Column Separations

A. Florisil Column:

1. Fraction I (6% ethyl ether containing 2% ethanol, 94% petroleum ether)

HCB, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, oxychlordane, heptachlor epoxide, gamma-chlordane, trans-nonachlor, toxaphene, PCB's, o,p'-DDE, alpha-Chlordane, p,p'-DDE, p,p'-DDT, cis-nonachlor, o,p'-DDT, p,p'-DDD, p,p'-DDT, mirex, dicofol, endosulfan I (Split with FII).

2. Fraction II (15% ethyl ether containing 2% ethanol, 85% petroleum ether)

dieldrin, endrin, dacthal, endosulfan I (split with FI), endosulfan II (split with FIII), endosulfan sulfate (split with FIII).

3. Fraction III (50% ethyl ether containing 2% ethanol, 50% petroleum ether)

endosulfan II (split with FII), endosulfan sulfate (split with FII), malathion.

B. Florisil Mini-Column:

1. Fraction I (12 ml hexane followed by 12 ml 1% methanol in hexane)

HCB, gamma-BHC (25%), alpha-BHC (splits with FII), trans-nonachlor, o,p'-DDE, p,p'-DDE, o,p'-DDD, p,p'-DDD (splits with FII), o,p'-DDT, p,p'-DDT, mirex, cis-nonachlor, cis-chlordane, trans-chlordane, PCB's

2. Fraction II (24 ml 1% methanol in hexane)

gamma BHC (75%), beta-BHC, alpha-BHC (splits with FI), delta-BHC, oxychlordane, heptachlor epoxide, toxaphene, dicofol, dacthal.

C. Silica Gel:

1. SG Fraction I (100 ml petroleum ether)

n-dodecane, n-tridecane, n-tetradecane, ocylcyclohexane, n-pentadecane, nonycyclohexane, n-hexadecane, n-heptadecane, pristane, n-octadecane, phytane, n-nonadecane, n-eicosane.

2. SG Fraction II (100 ml 40% methylene chloride in petroleum ether followed by 50 ml methylene chloride)

napthalene, fluorene, phenanthrene, anthracene, fluoranthrene, pyrene, 1,2-benzanthracene, chrysene, benzo [b] fluoranthrene, benzo [k] fluoranthrene, benzo [e] pyrene, benzo [a] pyrene, 1,2:5,6-dibenzanthracene, benzo [g,h,i] perylene.

D. Silicic Acid:

1. SA Fraction I (20 ml petroleum ether)

HCB, mirex

2. SA Fraction II (100ml petroleum ether)

PCB's, p,p'-DDE (splits with SA III)

3. SA Fraction III (20 ml mixed solvent: 1% acetonitrile, 80% methylene chloride, 19% hexane)

alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, oxychlordané, heptachlor epoxide, gamma-chlordane, trans-chlordane, toxaphene, o,p'-DDE, alpha-chlordane, p,p'-DDE (splits with SAII), o,p'-DDT, cis-nonachlor, o,p'-DDT, p,p'-DDD, p,p'-DDT, dicofol.

Method 6. Analysis For Chlorinated Hydrocarbon Pesticides And Related Compounds - Micro Method

This method is necessary when sample size is limited (below 4 g, approximately) and in case of organ tissue as substrate and is a modified version of the method described in Manual of Analytical Methods for the Analysis of Pesticides In Humans and Enviromental Samples, EPA-600/8-80-038, June 1980, Section 5, A (2). It is suitable for adipose, kidney, liver, muscle, brain, and other tissues:

1. Weigh 0.5 g or less of well-mixed tissue into a size 22 Duall tissue grinder.
2. Extract tissue by grinding three times with acetonitrile; the first time being with 4 ml followed by two 2.5 ml portions.
3. Remove the pestle after each grinding and centrifuge, decanting the extract into a 50 ml glass stoppered graduated mixing cylinder.
4. Combine all extracts and record the total volume of the three extracts.
5. Add a volume of PRQ water equivalent to 3.3 times the extract volume. Then add 2 ml saturated NaCl solution.
6. Extract the aqueous acetonitrile mixture with 5 ml hexane by vigorous shaking for 1 minute.
7. Allow layers to separate, and remove the hexane layer with a Pasteur pipet into a 15 ml screw-capped culture tube.
8. Re-extract twice with 2 ml hexane each time, combining the extracts into the culture tube.
9. Concentrate the combined hexane extracts under nitrogen to approximately 0.5 ml volume.
10. Clean-up on a florisil mini-column as described in Method 2, Steps 8, 9, 10. and 11.

Note For brain tissue additional treatment is necessary before column clean-up:

11. Proceed through Steps 1-9 above, add 0.3 ml acetic anhydride and 0.3 ml pyridine, cap tightly and incubate for 30 minutes in a water bath

at 60-65°C.

12. Add 8 ml PRQ water and 1 ml saturated NaCl and extract three times with 2 ml hexane, combining the extracts into a clean tube.
13. Concentrate the combined extracts under nitrogen to about 0.3 ml and proceed with florisil mini-column clean-up. (Step 10)

Note The following changes in sample handling, particularly column clean-up, should be observed for Kepone analysis:

14. Maintain the integrity of the analyte in sample extracts by insuring that the samples are not allowed to reach dryness during concentration steps. Kepone easily adheres to glass, but the use of polar solvents such as methanol and acetonitrile within the analysis will provide better recoveries of this analyte.
15. Modifications to florisil mini-column clean-up are as follows:
 - * Following addition of sample to the column, apply a 1ml rinse of 1% methanol in hexane to the sample tube. This rinse should be added after the first phase of the first fraction (12mls hexane) and will insure removal of trace quantities of kepone adhered to glass. Decrease the total volume of the second phase of the first fraction (12mls 1% methanol/hexane) to 11mls.
 - * Modify the total volume of the second fraction from 24mls to 36mls 1% methanol/hexane. This fraction contains Kepone.
 - * Concentrate column fractions on N-EVAP and transfer with 1% methanol/hexane to calibrated test tubes. Adjust sample volume to calibrated level and proceed to determination by gas chromatograph.

UNIVERSITY OF SOUTH ALABAMA GEOCHEMISTRY LABORATORY
ORGANIC CARBON-CARBONATE CARBON DETERMINATION

Reagents:

1. Acid solution (6N HCl). Dilute HCl 1:1 with deionized water passed through organic-scrub filter.
2. Buffer solution (pH 10). Dissolve 67.5 grams of ammonium chloride in 200 ml deionized, organic-scrubbed water; add 570 ml reagent grade concentrated ammonium hydroxide, and dilute to 1 liter.
3. Potassium cyanide solution (2% solution). Dilute 10 grams in 500 ml of deionized, organic-scrubbed water.
4. Eriochrome Black-T indicator solution. Dissolve 0.2 grams of the indicator powder (Eastman Kodak, P6361) in 500 ml of analytical reagent grade methanol or triethanolamine containing 2 grams of hydroxylamine hydrochloride.
5. EDTA solution. Dissolve 4 grams of EDTA (disodium dihydrogen ethylenediamine tetraacetate) in one liter of deionized, organic-scrubbed water.

Standardize:

Standardize these reagents against U.S. Bureau of Standards Dolomitic Limestone 88B which contains a known amount of total soluble alkaline earths.

Determination of Carbonate Carbon

(Turekian, K.K., 1956, Rapid technique for determination of carbonate content of deep-sea cores: Am. Assoc. Petrol. Geol. Bull. 10:2507-2509)

Procedure

Sample Preparation:

1. Weigh 20 mg (0.020 g) of finely ground sediment and place in a 125 ml Erlenmeyer flask. Label flask with Sample ID and weight.
2. Add about 5 ml of acid solution and heat on a hot plate (in fume hood) until completely dry. Remove flask from heat and allow to cool.
3. Add approximately 1 ml of acid solution to flask to dissolve soluble material.
4. Dilute with 50 ml of deionized, organic-scrubbed water.
5. Add 5 ml of the buffer solution.

6. Add 2 ml of potassium cyanide and 10 drops of indicator. The sample should be some shade of purple (or possibly blue).

Titration

1. Record the volume of EDTA solution (to nearest 0.5 ml) in the burette prior to beginning titration.
2. Place a clean magnet in the flask and place flask on illuminated magnetic stirrer.
3. Titrate sample solution with EDTA. The end point occurs with a change in color from "wine red" to "true blue."

NOTE:

The color of the original sample solution is dependent upon the amount of soluble alkaline earths present in each sediment sample. For this reason, the color of the sample solution can be wine red (for a sample with abundant soluble alkaline earths), bluish-purple (for a sample with moderate amounts), or blue (for a sample with none, or trace amounts only). The end point of the titration is not always a rich blue, but may be light blue or even gray. Regardless, the end point will be easy to identify.

4. When the presumed end point is reached, note the burette volume and then add another drop (or more) to see if further color change occurs. If a change does occur, record the new volume.
5. Remove the magnet from the flask and wash with deionized, organic-scrubbed water; place in next sample to be analyzed.
6. When analysis is complete, dispose of solutions according to posted laboratory instructions.

Calculations:

The percent calcium carbonate is calculated by the following formula:

$$\% \text{CaCO}_3 = (\text{standardization constant}) \times (\text{titration volume}) / \text{Sample Wt} \times$$

* sample weight in milligrams

$$\% \text{Carbonate Carbon} = (\% \text{CaCO}_3) \times (0.12)$$

Discussion:

The standard solution is standardized in terms of millimoles of alkaline-earth per ml of solution. Stoichiometrically this must be equivalent to the millimoles of CO_3^{2-} . Hence, it is possible to derive the CaCO_3 content. Two basic assumptions are used:

- 1- all of the soluble alkaline earths are present as carbonates and all carbonate is bound to the alkaline earths
- 2- no measureable alkaline earths bound to the silicate fraction are released with dilute HCl treatment.

Organic Carbon Determination

To obtain a value for organic carbon, the percentage of carbonate carbon in the sample is subtracted from the percent total carbon. Total carbon percent is determine using the LECO induction furnace.

Total Carbon Analysis

1. Extract 1 - 2 grams of sample from freshly collected grab or core sample.
2. Place on 2.5 cm plastic tray and dry in 50 C oven for 24 hours.
3. Grind sample in agate mortar and pestle and sieve through 60 mesh screen (avoid touching sample with hands).
4. Transfer sample to LECO crucible and store in dessicator until needed.

LECO Procedure:

- 1 - Condition the LECO absorption bulb by running at least three steel ring standards (one ring standard and one scoop of copper accelerator should be placed in each crucible). Alpha-cel Tin Coated Standards*, containing 0.876 % carbon (plus or minus 0.008%), or equivalent, are recommended.

*Available from Alpha Resources, Inc.
Box 199, Stevensville, MI 49127
(616) 465-5559

NOTE:

The absorption bulb is weighed to the nearest 0.00001 g before and after combustion. The weight increase is due to absorption of CO₂. The practical lower limit of this technique is 0.01% Carbon. The upper limit can be extended to 30% carbon by reducing the sample size. The accuracy is (plus or minus) 0.005% (or plus or minus 1% of the carbon present, whichever is greater).

- 2 - When the absorption bulb has been conditioned (and steel ring standard weights are determined within experimental limits), samples can then be run by adding one scoop of copper accelerator to each crucible and then carrying out the standard LECO procedure. A steel ring standard should be run at least every 10 samples to preserve quality assurance.
- 3 - Percent Total Carbon is calculated from the following formula:

$$\% \text{ Carbon} = (B \times 0.2729 \times 100) / W$$

where B = the weight gain of the bulb due to CO₂ absorpction
and W = the weight of the sample

NOTE: (0.2729 is the ratio of C / CO₂)

4 - Percent Organic Carbon is then determined by difference:

% Organic Carbon = % Total Carbon - % Carbonate Carbon

APPENDIX FOUR

STANDARD OPERATING

PROCEDURES

FOR

COLLECTION OF FISH

SAMPLING PROCEDURES

STANDARD OPERATING PROCEDURES**COLLECTION OF FISH TISSUE SAMPLES**

Fish collected for chemical contaminant evaluations may be taken by electrofishing gear, monofilament gillnets, otter trawl, haul or beach seines, fish traps, trotlines, or rod and reel. However, any collecting gear should be free of chemical treatments and/or metals that could contaminate samples. This is particularly important when the entire fish (whole body analysis) will be used.

For species of special concern such as Gulf sturgeon or large broodstock striped bass, we utilize only incidental mortalities, and these should be fresh specimens.

The following is a sample dissection.

1. Wash hands thoroughly and rinse completely. Wear vinyl or latex gloves. Final rinse with distilled water.
2. Fish should be clean. It may be rinsed of debris or mud in the waters of the collection site.
3. The dissection surface (work area) should be a chemically inert substance such as a stainless steel acetone-rinsed pan, or counter. Avoid letting the dissected sample touch this surface, if possible.
4. Use previously cleaned, and acetone rinsed, then distilled water-rinsed stainless steel dissection tools (knives, scalpels, etc.). Scales for total fish weights and sample weights should also be clean or covered with pre-cleaned aluminum foil. Measuring devices for fish lengths, etc., should be clean, or should not come in contact with the specimen.
5. Do not let dissected samples remain exposed to the air. Exposure can dry samples and reduce the natural percentage of moisture. Prepare each dissected sample for shipping or freezing as it is dissected.
6. Samples should be placed in the smallest, pre-cleaned glass jar that will adequately hold the sample. The jars should be pre-labeled with a permanent, waterproof marking pen on the outside of the jar. Jars should also have a teflon liner inside the lid. As an alternative, acetone-rinsed, heavy-duty aluminum foil may be used to wrap the sample. After double-wrapping, place the sample (with sample identification label) inside an air-tight zip-lock bag.

7. Sample identification labels should be prepared with permanent, waterproof ink or other writing instruments that will not bleed out or wash out, and should provide the following information:
 - a. species name and common name,
 - b. type of tissue (if not whole body),
 - c. collection location,
 - d. latitude and longitude,
 - e. county and state,
 - f. weight of sample in grams,
 - g. date of collection,
 - h. sample collector's name,
 - i. total weight of fish specimen (grams),
 - j. total length and fork length of specimen (cm), and
 - k. method of collection.
8. Samples should be frozen as soon as possible. If samples contain large amounts of liquids that may expand, the lids may be set on the jars, without securing, until the sample has expanded and frozen. Then lids should be secured tightly.
9. Photographs of the specimens are desirable, as well as a written description of any external or internal lesions, tumors, etc.

APPENDIX FIVE

**HURRICANE AND
TROPICAL STORM
OCCURRENCES**

APPENDIX 5

Appendix 5. Hurricanes and tropical storms occurring within 90 miles of St. Joseph Bay, Florida, between 1871 and 1995. Storm categories for maximum sustained wind speeds (miles/hour) are: 1 = 74-95; 2 = 96-110; 3 = 111 - 130 (Saffir/Simpson Hurricane Scale).

Year	Date	Name	Storm #	Storm Type	Category	20-mile rng	Over Bay
1871	Oct 5	none	6	U	U	X	
1873	Sept 19	none	3	U	U		
1875	Sept 27	none	3	U	U		X
1877	Sept 19	none	2	U	U		X
1877	Oct 2	none	4	U	U		X
1878	Oct 10	none	6	U	U		X
1880	Aug 30	none	4	U	U		X
1885	Aug 30	none	3	U	U	X	
1885	Sept 21	none	4	U	U		X
1886	June 21	none	2	H	U	X	
1887	July 27	none	2	H	U		
1894	Oct 8	none	4	H	U		X
1896	July 7	none	1	H	U		
1898	Aug 2	none	1	H	U		X
1899	Aug 1	none	1	H	U	X	
1901	Sept 27	none	8	TS	U		
1902	June 14	none	1	TS	U		
1903	Sept 13	none	3	H	2		X ¹
1906	June 12	none	1	TS	-		X
1907	June 28	none	1	TS	-	X	
1907	Sept 28	none	3	TS	-	X	
1909	June 29	none	2	TS	-		
1915	Sept 4	none	4	H	1		X
1917	Sept 28	none	3	H	3		
1924	Sept 15	none	4	H	1		X
1928	Aug 14	none	2	TS	-	X	
1929	Sept 30	none	2	H	3		X

Appendix 5 continued.

Year	Date	Name	Storm #	Storm Type	Category	20-mile rng	Over Bay
1933	Aug 20	none	6	TS	-		
1936	Aug 30	none	5	H	3		
1937	Sept 20	none	6	TS	-	X	
1938	Oct 23	none	7	TS	-		
1939	Aug 12	none	2	H	1		X
1941	Oct 6	none	5	H	2		
1948	July 8	none	2	TS	-		
1953	June 6	Alice	1	TS	-		
1953	Sept 19	none	7	TS	-		
1953	Sept 26	Florence	8	H	1		
1956	Sept 24	Flossy	7	H	2		
1957	June 8	none	1	TS	-		
1957	Sept 8	Debbie	5	TS	-		
1965	June 15	none	1	TS	-		
1966	June 9	Alma ²	1	H	2		
1970	July 22	Becky	2	TS	-		X
1972	June 19	Agnes	2	H	1		X
1975	Sept 23	Eloise ³	5	H	3		
1985	Nov 21	Kate	11	H	2		X
1987	Aug 15	none	1	TS	-		X
1994	Aug 15	Beryl	2	TS	-		
1995	Oct 4	Opal ⁴	15	H	3		

¹ Although this storm was a hurricane as it initially made landfall north of Miami, Florida, upon crossing the peninsula of Florida and moving into the Gulf of Mexico, it made not have re-intensified beyond that of tropical storm strength before crossing over St. Joseph Bay.

² Alma made landfall near the mouth of the Ochlockonee River, approx. 50 miles east of St. Joseph Bay.

³ Eloise made landfall in eastern Walton County, Florida, approximately 42 miles west of St. Joseph Bay.

⁴ Opal made landfall near Navarre Beach, Florida, approximately 85 miles west of St. Joseph Bay. However, Opal was one of the largest hurricanes of record, as far as the width of coastline affected. Extensive damage occurred between Pensacola Beach and Mexico Beach, Florida, a stretch of coastline approximately 120 miles in length.

APPENDIX SIX

SEDIMENT DATA

FOR

ST. JOSEPH BAY

APPENDIX 6

Appendix 6 . Sediment Data Base for St. Joseph Bay, Florida

page 1

St.#	Other ID	Latitude	Longitude	Depth (feet)	Depth (meters)	% TOC	% CaCO3	% Gravel	% Sand	% Silt	% Clay	% Unkn	Sample Date
	SJA1	29°53.00	85°24.00	29.0	8.8	4.11	3.19	0.33	11.65	39.59	48.42	0.00	12-Jun-91
	SJA4-1	29°50.01	85°24.36	6.0	1.8	0.12	0.43	0.00	98.97	0.00	0.00	1.03	12-Jun-91
	SJA4-2	29°49.69	85°24.43	30.0	9.1	0.50	2.66	0.00	59.34	11.20	29.46	0.00	13-Jun-91
	SJA5	29°48.98	85°24.00	36.0	11.0	4.81	3.49	0.00	13.36	35.75	50.88	0.00	13-Jun-91
	SJA5-1	29°48.49	85°24.29	33.0	10.1	2.10	2.14	0.00	6.70	37.51	55.79	0.00	13-Jun-91
	SJA6	29°48.01	85°23.99	31.0	9.4	4.52	0.31	9.29	8.22	31.73	50.76	0.00	13-Jun-91
	SJA6-1	29°47.91	85°24.23	6.0	1.8	0.03	0.09	0.00	99.36	0.00	0.00	0.64	13-Jun-91
	SJA7	29°46.99	85°24.02	4.0	1.2	0.00	1.23	1.45	98.27	0.00	0.00	0.28	13-Jun-91
	SJA7-1	29°46.31	85°23.99	17.0	5.2	4.54	0.27	0.43	99.08	0.00	0.00	0.49	12-Jun-91
	SJA8-1	29°45.84	85°24.15	4.5	1.4	0.68	0.11	0.00	99.09	0.00	0.00	0.91	12-Jun-91
	SJB1	29°53.01	85°23.01	38.0	11.6	0.26	5.1	5.74	93.39	0.00	0.00	0.86	12-Jun-91
	SJB2	29°52.03	85°23.00	36.0	11.0	1.92	1.99	0.00	29.74	22.31	47.95	0.00	12-Jun-91
	SJB2-1	29°51.99	85°23.58	6.0	1.8	0.07	1.47	0.00	98.75	0.00	0.00	1.25	12-Jun-91
	SJB3	29°51.00	85°22.98	36.0	11.0	4.38	3.01	0.00	6.06	35.25	58.69	0.00	12-Jun-91
	SJB3-1	29°50.81	85°23.00	35.0	10.7	0.58	3.04	0.58	75.15	7.59	16.68	0.00	12-Jun-91
	SJB4	29°50.01	85°23.01	36.0	11.0	1.92	3.99	0.00	17.48	23.77	58.75	0.00	12-Jun-91
	SJB5	29°49.00	85°23.01	35.0	10.7	0.54	3.44	9.29	9.57	31.38	49.76	0.00	13-Jun-91
	SJB6	29°47.99	85°22.99	33.0	10.1	0.01	2.86	3.39	13.06	32.17	51.38	0.00	13-Jun-91
	SJB7	29°46.99	85°23.03	28.0	8.5	2.16	3.51	3.08	17.15	39.48	40.30	0.00	13-Jun-91
	SJB7-1	29°47.00	85°23.79	6.0	1.8	0.05	0.14	0.00	99.26	0.00	0.00	0.74	13-Jun-91
	SJB7-2	29°46.44	85°23.73	24.0	7.3	0.96	3.83	0.35	73.45	6.89	19.30	0.00	12-Jun-91
	SJB8	29°45.98	85°23.02	28.0	8.5	1.65	2.12	0.00	48.33	16.52	35.15	0.00	13-Jun-91
	SJB8-1	29°45.98	85°23.46	7.0	2.1	0.04	0.2	0.76	98.07	0.00	0.00	1.16	13-Jun-91
	SJB9	29°44.99	85°23.02	24.0	7.3	1.99	3.88	0.49	33.01	32.40	34.11	0.00	13-Jun-91
	SJB9-1	29°44.12	85°23.26	8.0	2.4	0.74	0.25	0.00	98.62	0.00	0.00	1.38	13-Jun-91
	SJC1	29°53.01	85°22.00	26.0	7.9	0.57	1.59	2.66	96.51	0.00	0.00	0.83	12-Jun-91
	SJC2	29°52.02	85°22.00	28.0	8.5	0.82	2.95	4.10	76.87	5.46	0.00	13.58	12-Jun-91
	SJC3	29°51.01	85°22.00	31.0	9.4	0.78	4.3	3.98	71.41	7.39	17.22	0.00	12-Jun-91
	SJC3-1	29°50.41	85°21.97	35.0	10.7	2.92	3.41	0.00	22.79	18.08	59.13	0.00	12-Jun-91

St.#	Other ID	Latitude	Longitude	Depth (feet)	Depth (meters)	% TOC	% CaCO ₃	% Gravel	% Sand	% Silt	% Clay	% Unkn	Sample Date
	SJC4	29°50.01	85°22.01	34.0	10.4	0.63	1.88	0.76	97.62	0.00	0.00	1.62	12-Jun-91
	SJC5	29°48.98	85°21.98	42.0	12.8	4.49	3.22	0.00	21.66	35.37	42.97	0.00	13-Jun-91
	SJC6	29°47.99	85°22.02	40.0	12.2	3.89	3.24	4.82	18.31	33.33	43.54	0.00	13-Jun-91
	SJC7	29°46.98	85°22.01	38.0	11.6	1.17	3.89	0.00	42.21	22.65	35.14	0.00	13-Jun-91
	SJC8	29°46.00	85°22.03	25.0	7.6	5.94	3.19	2.41	19.76	35.85	41.98	0.00	13-Jun-91
	SJC9	29°44.98	85°22.02	25.0	7.6	5.31	1.57	1.21	98.07	0.00	0.00	0.72	13-Jun-91
	SJC10	29°44.00	85°22.02	18.0	5.5	0.54	2.91	5.23	94.23	0.00	0.00	0.54	13-Jun-91
	SJC11	29°43.02	85°22.00	12.0	3.7	0.14	0.27	0.00	98.98	0.00	0.00	1.02	11-Jun-91
	SJC11-1	29°42.58	85°22.65	3.0	0.9	na	na	1.29	97.34	0.00	0.00	1.37	13-Jun-91
	SJC11-2	29°41.94	85°22.65	2.0	0.6	na	na	0.27	98.16	0.00	0.00	1.58	13-Jun-91
	SJC12-1	29°41.70	85°22.34	3	0.9	na	na	3.29	94.71	0.00	0.00	2.00	13-Jun-91
	SJD2	29°52.01	85°21.01	22.0	6.7	0.90	2.46	4.64	91.68	0.00	0.00	3.68	12-Jun-91
	SJD3	29°51.03	85°20.98	27.0	8.2	1.10	3.55	0.26	55.21	12.06	32.47	0.00	12-Jun-91
	SJD4	29°50.01	85°21.03	31.0	9.4	3.94	2.56	0.00	20.82	17.32	61.86	0.00	12-Jun-91
	SJD5	29°49.03	85°21.04	31.5	9.6	4.55	3.48	0.79	20.03	34.35	44.83	0.00	12-Jun-91
	SJD6	29°48.05	85°20.99	30.0	9.1	0.07	3.11	0.00	24.53	35.44	40.03	0.00	11-Jun-91
	SJD7	29°47.05	85°21.03	30.0	9.1	4.63	2.38	2.02	19.85	37.49	40.64	0.00	11-Jun-91
	SJD8	29°46.04	85°21.00	31.0	9.4	5.17	3.31	0.00	9.80	44.77	45.43	0.00	11-Jun-91
	SJD9	29°45.01	85°22.03	24.0	7.3	1.93	3.57	2.02	66.77	7.54	23.67	0.00	11-Jun-91
	SJD10	29°44.00	85°21.00	12.0	3.7	0.58	3.98	2.30	96.71	0.00	0.00	0.99	11-Jun-91
	SJD10-1	29°43.08	85°21.95	21.0	6.4	0.50	1.58	0.83	97.47	0.00	0.00	1.69	11-Jun-91
	SJD11	29°43.00	85°21.03	6.0	1.8	0.08	0.17	0.00	99.37	0.00	0.00	0.63	11-Jun-91
	SJD11-1	29°42.71	85°21.51	12.0	3.7	0.38	0.99	2.17	97.41	0.00	0.00	0.42	11-Jun-91
	SJD11-2	29°42.13	85°21.91	20.0	6.1	0.25	3.44	0.00	99.74	0.00	0.00	0.26	11-Jun-91
	SJD12	29°41.99	85°21.01	3.0	0.9	4.27	0.12	0.00	96.99	0.00	0.00	3.01	11-Jun-91
	SJD12-1	29°41.84	85°21.43	12.0	3.7	0.90	1.56	1.79	95.45	0.00	0.00	2.75	11-Jun-91
	SJD12-2	29°42.02	85°21.93	20.0	6.1	4.14	1.12	0.31	58.39	16.15	25.16	0.00	11-Jun-91
	SJE2-1	29°51.99	85°20.81	7.0	2.1	0.20	0.97	0.00	99.71	0.00	0.00	0.29	12-Jun-91
	SJE4	29°50.02	85°20.04	24.0	7.3	1.98	2.96	0.00	10.19	30.53	59.28	0.00	10-Jun-91
	SJE4-1	29°49.54	85°20.03	24.0	7.3	1.57	2.87	0.00	41.13	17.36	41.51	0.00	10-Jun-91

Appendix 6 cont'd. Sediment Data Base for St. Joseph Bay, Florida

page 3

St.#	Other ID	Latitude	Longitude	Depth (feet)	Depth (meters)	% TOC	% CaCO ₃	% Gravel	% Sand	% Silt	% Clay	% Unkn	Sample Date
	SJE5	29°48.99	85°20.01	24.0	7.3	3.28	2.59	0.00	6.84	38.99	54.17	0.00	10-Jun-91
	SJE6	29°48.01	85°20.06	26.0	7.9	1.69	3.21	0.00	10.19	30.05	59.76	0.00	10-Jun-91
	SJE7	29°47.01	85°20.02	25.0	7.6	3.57	3.38	3.33	22.70	36.00	37.96	0.00	10-Jun-91
	SJE8	29°45.98	85°20.00	22.0	6.7	0.95	3.67	11.54	63.78	8.41	16.27	0.00	10-Jun-91
	SJE9	29°45.01	85°20.07	19.0	5.8	0.57	1.62	10.38	88.12	0.00	0.00	1.50	10-Jun-91
	SJE10	29°44.01	85°20.00	2.5	0.8	na	na	0.26	98.91	0.00	0.00	0.82	10-Jun-91
	SJE10-1	29°44.00	85°20.12	7.0	2.1	na	na	0.20	98.94	0.00	0.00	0.86	10-Jun-91
	SJE10-2	29°43.16	85°20.00	20.0	6.1	0.56	0.63	4.05	95.46	0.00	0.00	0.49	11-Jun-91
	SJE11	29°43.04	85°19.98	8.0	2.4	0.12	0.31	0.00	99.53	0.00	0.00	0.47	11-Jun-91
	SJE11-1	29°42.67	85°20.03	12.0	3.7	1.04	1.43	1.88	97.39	0.00	0.00	0.73	13-Jun-91
	SJE11-2	29°42.82	85°20.52	16.0	4.9	0.28	0.23	0.00	98.79	0.00	0.00	1.21	11-Jun-91
	SJE12	29°41.99	85°20.03	10.0	3.0	0.07	0.28	0.00	99.25	0.00	0.00	0.75	11-Jun-91
	SJF4	29°50.01	85°19.07	7.0	2.1	2.04	0.23	0.45	53.36	17.42	28.76	0.00	10-Jun-91
	SJF4-1	29°49.27	85°19.01	5.0	1.5	0.17	1.52	6.10	93.20	0.00	0.00	0.70	10-Jun-91
	SJF5	29°49.02	85°19.01	5.0	1.5	0.60	1.47	2.70	96.53	0.00	0.00	0.77	10-Jun-91
	SJF6	29°47.98	85°19.01	21.0	6.4	0.84	5.43	74.87	23.37	0.00	0.00	1.76	07-Jun-91
	SJF7	29°46.95	85°19.00	19.0	5.8	1.06	1.65	6.58	90.15	0.00	0.00	3.26	07-Jun-91
	SJF8	29°45.99	85°19.00	14.5	4.4	0.72	1.96	4.25	93.71	0.00	0.00	2.04	07-Jun-91
	SJF9	29°44.98	85°19.01	5.0	1.5	0.06	1.7	1.42	98.05	0.00	0.00	0.53	07-Jun-91
	SJF10	29°43.99	85°18.99	3.0	0.9	2.36	0.77	0.74	96.59	0.00	0.00	2.68	11-Jun-91
	SJF11	29°42.98	85°19.03	3.0	0.9	0.00	2.11	1.06	98.54	0.00	0.00	0.40	11-Jun-91
	SJG6-1	29°48.00	85°18.32	6.0	1.8	0.18	1.55	0.65	98.68	0.00	0.00	0.67	07-Jun-91
	SJG8-1	29°45.98	85°18.82	6.0	1.8	0.06	1.23	0.36	99.20	0.00	0.00	0.44	07-Jun-91
	SJG8-2	29°45.89	85°18.38	2.0	0.6	na	na	0.03	97.58	0.00	0.00	2.39	13-Jun-91
	SJG8-3	29°44.98	85°18.30	4.2	1.3	0.80	0.05	0.19	94.85	0.00	0.00	4.96	13-Jun-91
	SJG9-1	29°44.87	85°18.38	6.0	1.8	0.17	1.12	0.41	98.48	0.00	0.00	1.11	13-Jun-91
	SJH6-1	29°47.92	85°17.98	2.0	0.6	na	na	0.23	99.01	0.00	0.00	0.76	13-Jun-91

St.#	Other ID	Latitude	Longitude	Depth (feet)	Depth (meters)	% TOC	% CaCO ₃	% Gravel	% Sand	% Silt	% Clay	% Unkn	Sample Date
	SJH6-2	29°47.90	85°18.08	3.0	0.9	na	na	9.16	88.85	0.00	0.00	1.99	13-Jun-91
	D3	29°51.00	85°21.00	26.0	7.9	3.49	3.38	5.52	45.96	24.51	24.01	0.00	11-Jun-91
	D6	29°48.00	85°21.00	30.0	9.1	5.33	1.73	0.00	4.25	52.11	43.64	0.00	11-Jun-91
	Milldock	29°48.95	85°18.85	30.0	9.1	6.86	1.55	5.49	29.50	25.58	39.43	0.00	11-Jun-91
	SJ1	29°53.19	85°15.07	17.0	5.2	0.28	0.03	0.00	98.72	0.00	0.00	1.28	02-Feb-91
	SJ2	29°51.83	85°16.55	12.0	3.7	0.11	0.09	0.00	99.44	0.00	0.00	0.56	02-Feb-91
	SJ3	29°50.40	85°18.10	4.0	1.2	0.98	15.1	5.55	21.40	33.94	39.11	0.00	02-Feb-91
	SJ4	29°50.18	85°18.46	14.0	4.3	0.09	0.07	0.00	99.80	0.00	0.00	0.20	02-Feb-91
	SJ5	29°49.98	85°18.67	4.0	1.2	3.05	0.23	0.00	81.70	6.42	0.00	11.88	02-Feb-91
	SJ6	29°49.7	85°19.24	10.0	3.0	1.36	0.73	0.00	77.18	9.60	0.00	13.22	02-Feb-91
	SJ7	29°49.8	85°19.56	17.0	5.2	5.95	1.61	0.00	37.56	31.99	30.45	0.00	02-Feb-91
	SJ8	29°49.94	85°19.41	10.0	3.0	0.71	1.19	2.33	93.31	0.00	0.00	4.36	02-Feb-91
	SJ9	29°50.07	85°19.24	7.0	2.1	0.50	0.99	0.00	97.97	0.00	0.00	2.03	02-Feb-91
	SJ10	29°50.12	85°19.18	2.0	0.6	0.17	0.1	0.00	99.24	0.00	0.00	0.76	02-Feb-91
	SJ11	29°49.62	85°19.04	21.0	6.4	0.18	0.07	0.00	99.33	0.00	0.00	0.67	02-Feb-91
	SJ12	29°49.55	85°19.16	9.0	2.7	1.09	1.43	0.00	76.57	8.29	15.14	0.00	02-Feb-91
	SJ13	29°49.40	85°19.40	32.0	9.8	4.47	1.57	0.00	17.12	36.46	46.42	0.00	02-Feb-91
	SJ14	29°49.11	85°18.96	27.0	8.2	5.06	1.51	2.87	27.86	29.96	39.31	0.00	02-Feb-91
	SJ15	29°49.04	85°18.95	32.0	9.8	4.80	1.86	4.09	21.69	33.89	40.33	0.00	02-Feb-91
	SJ16	29°48.99	85°18.90	33.0	10.1	4.53	1.54	4.25	30.56	24.54	40.65	0.00	02-Feb-91
	SJ17	29°48.94	85°18.90	32.0	9.8	4.21	1.59	0.44	19.04	34.83	45.69	0.00	02-Feb-91
	SJ18	29°48.84	85°18.86	32.0	9.8	2.74	3.61	0.00	13.90	25.86	60.24	0.00	02-Feb-91
	SJ19	29°49.11	85°19.10	29.0	8.8	3.52	1.23	0.57	28.89	31.27	39.26	0.00	02-Feb-91
	SJ20	29°49.03	85°19.06	29.0	8.8	2.75	1.49	1.62	23.84	37.48	37.06	0.00	02-Feb-91
	SJ21	29°48.94	85°19.05	28.0	8.5	4.41	1.53	0.45	25.88	32.67	40.99	0.00	02-Feb-91
	SJ22	29°48.86	85°19.01	28.0	8.5	2.76	1.47	0.59	26.01	32.99	40.41	0.00	02-Feb-91
	SJ23	29°48.78	85°18.97	28.0	8.5	3.35	1.6	0.00	19.21	29.64	51.15	0.00	02-Feb-91